Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[2-(Dimethylamino)ethanol-κ^2N,O]-[2-(dimethylamino)ethanolato-κ^2N,O]-iodidocopper(II)

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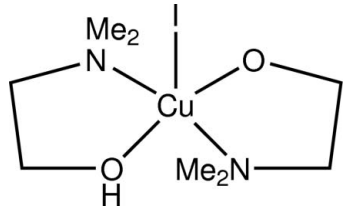
Received 15 February 2012; accepted 7 March 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.019 Å; R factor = 0.058; wR factor = 0.204; data-to-parameter ratio = 18.6.

The title compound, [Cu($C_4H_{10}NO$)I($C_4H_{11}NO$)], was obtained unintentionally as the product of an attempted synthesis of a Cu/Zn mixed-metal complex using zerovalent copper, zinc(II) oxide and ammonium iodide in pure 2-(dimethylamino)ethanol, in air. The molecular complex has no crystallographically imposed symmetry. The coordination geometry around the metal atom is distorted square-pyramidal. The equatorial coordination around copper involves donor atoms of the bidentate chelating 2-(dimethylamino)ethanol ligand and the 2-(dimethylamino)ethanolate group, which are mutually *trans* to each other, with four approximately equal short Cu—O/N bond distances. The axial Cu—I bond is substantially elongated. Intermolecular hydrogen-bonding interactions involving the –OH group of the neutral 2-(dimethylamino)ethanol ligand to the O atom of the monodeprotonated 2-(dimethylamino)ethanolate group of the molecule related by the *n*-glide plane, as indicated by the O···O distance of 2.482 (12) Å, form chains of molecules propagating along [101].

Related literature

For background to the synthesis, see: Vinogradova *et al.* (2002). Buvaylo *et al.* (2009, 2011). Elongation of the axial Cu—I bond is common in this kind of compound, see: Wells (1986).



Experimental

Crystal data

[Cu($C_4H_{10}NO$)I($C_4H_{11}NO$)]
M_r = 367.71
Monoclinic, $P2_1/n$
a = 8.690 (1) Å
b = 15.241 (1) Å
c = 11.116 (1) Å
β = 106.847 (10)°
V = 1409.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.72 mm^{-1}
T = 296 K
0.32 × 0.3 × 0.2 mm

Data collection

Rigaku AFC-6S diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.333, T_{max} = 0.47
2642 measured reflections
2471 independent reflections
1003 reflections with $I > 2\sigma(I)$
R_{int} = 0.089
3 standard reflections every 150 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.204
S = 1.00
2471 reflections
133 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

Table 1
Selected bond lengths (Å).

I1—Cu1	2.928 (2)	Cu1—N1	2.059 (10)
Cu1—O1	2.030 (9)	Cu1—O2	2.010 (8)
Cu1—N2	2.058 (11)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*O*···O1^{i}	0.82	1.68	2.482 (12)	167

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *AFC6S Diffractometer Control Software* (Molecular Structure Corporation, 1998); cell refinement: *AFC6S Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: Johnson (1976); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We thank Professor Philip J. Squattrito for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2178).

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supporting information

Acta Cryst. (2012). E**68**, m419–m420 [https://doi.org/10.1107/S1600536812010215]

[2-(Dimethylamino)ethanol-κ^2N,O][2-(dimethylamino)ethanolato-κ^2N,O]iodidocopper(II)

Elena A. Buvaylo, Volodymyr N. Kokozay, Olga Yu. Vassilyeva and Brian W. Skelton

S1. Comment

In our previous study metal powders of zinc and copper were found to react with ammonium iodide and 2-(dimethylamino)ethanol (HMe_2Ea) in methanol, in air, affording the new heterotrinuclear complex $[Cu_2Zn(NH_3)I_3(Me_2Ea)_3]$ (Vinogradova *et al.*, 2002). Reactions employing elemental copper and aminoalcohol allow *in situ* formation of the metal aminoalkoxo species - key building blocks that can subsequently self-assemble with other metal centres present in the reaction vessel (Buvaylo *et al.*, 2009; Buvaylo *et al.*, 2011). The title compound was isolated from the solution obtained by reacting copper powder and zinc oxide with ammonium iodide in pure 2-(dimethylamino)ethanol. It can be considered as an intermediate, a building block that failed self-organization with another metal species produced in the reaction medium. To the best of our knowledge the title compound has not been structurally characterized.

The molecular complex has no crystallographically imposed symmetry (Fig. 1). The coordination geometry around the metal atom is distorted square pyramidal. The equatorial coordination around Cu(1) involves donor atoms of bidentate chelating 2-(dimethylamino)ethanol and 2-(dimethylamino)ethanolato group, which are mutually *trans* to each other, with four approximately equal short distances. The axial Cu(1)–I(1) bond is substantially elongated [2.928 (2) Å], and it is common for this kind of compounds (Wells, 1986).

Intermolecular hydrogen-bonding interactions from the OH group of neutral 2-(dimethylamino)ethanol ligand to O atom of monodeprotonated 2-(dimethylamino)ethanolato group of the molecule related by the *n* glide plane [H2O···O1{*x* - 1/2, -*y* + 1/2, *z* - 1/2} = 1.68 Å; O2···O1{*x* - 1/2, -*y* + 1/2, *z* - 1/2} = 2.482 (12) Å and O2—H2O···O1{*x* - 1/2, -*y* + 1/2, *z* - 1/2} = 167.3°] form chains of $Cu(Me_2Ea)(HMe_2Ea)I$ molecules propagated along the 101 direction (Fig. 2). Cu···Cu separations in the crystal are > 6.6 Å.

S2. Experimental

Copper powder (0.16 g, 2.5 mmol), ZnO (0.20 g, 2.5 mmol), NH_4I (1.45 g, 10 mmol), HMe_2Ea (15 ml) were heated to 323–333 K and magnetically stirred until total dissolution of the copper and ZnO was observed (95 min). The resulting blue solution was filtered and allowed to stand at room temperature. Green-blue microcrystals of the title compound were formed after one day. They were collected by filter-suction, washed with dry Pr^iOH and finally dried *in vacuo* (yield: 0.23 g).

S3. Refinement

The non-hydrogen atoms were refined anisotropically. The hydrogen atom on O2 atom was located and its position constrained with the isotropic displacement parameter allowed to refine. Other hydrogen atoms were placed at idealized positions (C–H = 0.95 Å, U_{iso}H = 1.20U_{eq} C for CH_2, 1.5U_{eq} C for CH_3) and not refined.

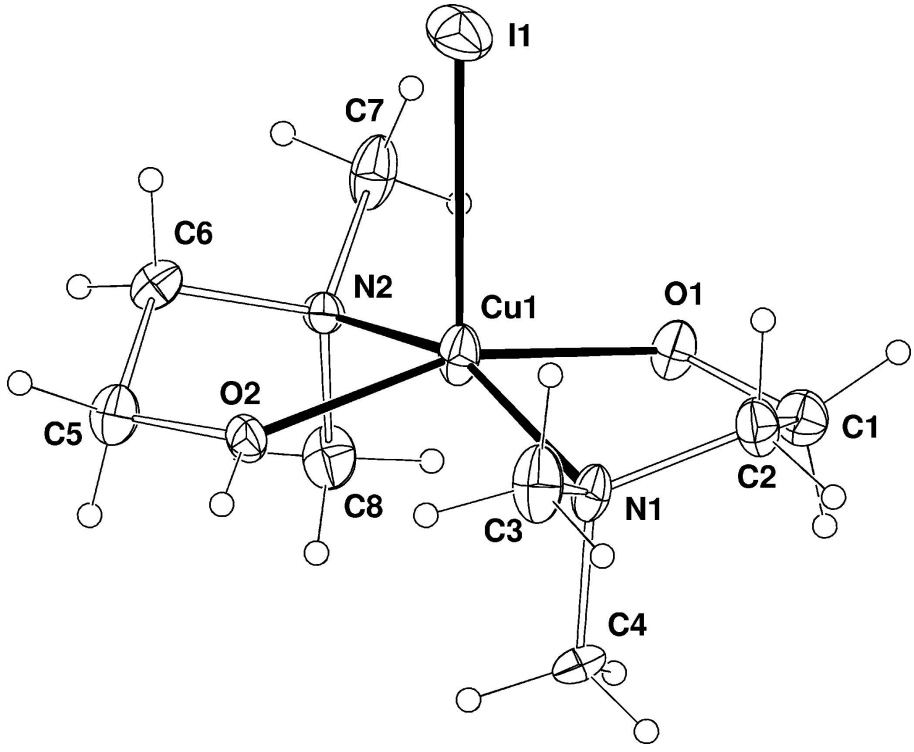

Figure 1

Molecular structure of the complex with the numbering scheme (the non-hydrogen atoms shown as 20% thermal ellipsoids).

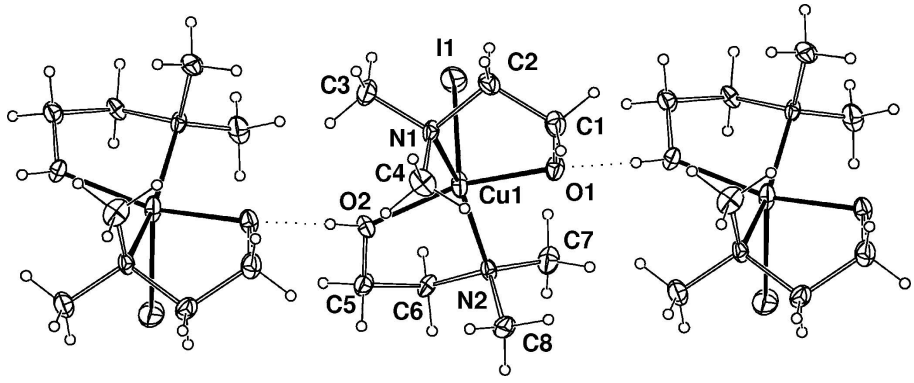

Figure 2

Hydrogen-bonding interactions between $Cu(Me_2Ea)(HMe_2Ea)I$ molecules within a polymeric chain.

[2-(Dimethylamino)ethanol-κ^2N,O][2-(dimethylamino)ethanolato-κ^2N,O]iodidocopper(II)

Crystal data

$[Cu(C_4H_{10}NO)I(C_4H_{11}NO)]$
M_r = 367.71
Monoclinic, $P2_1/n$
Hall symbol: -p 2yn
a = 8.690 (1) Å
b = 15.241 (1) Å
c = 11.116 (1) Å
β = 106.847 (10)°
V = 1409.1 (2) Å^3
Z = 4
$F(000)$ = 724
D_x = 1.733 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71069 Å
Cell parameters from 6 reflections

θ = 10.9–11.9°
μ = 3.72 mm^{-1}
T = 296 K

Rod, blue-green
0.32 × 0.3 × 0.2 mm

Data collection

Rigaku AFC-6S diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
2θ–ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.333, T_{max} = 0.47
2642 measured reflections

2471 independent reflections
1003 reflections with $I > 2\sigma(I)$
R_{int} = 0.089
θ_{max} = 25°, θ_{min} = 2.3°
h = 0→10
k = 0→18
l = −13→12
3 standard reflections every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.204
S = 1.00
2471 reflections
133 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0962P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.013
$\Delta\rho_{max}$ = 1.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.83 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
I1	0.64794 (15)	0.39539 (8)	0.25072 (11)	0.0679 (5)
Cu1	0.4723 (2)	0.26167 (12)	0.34694 (15)	0.0456 (6)
O1	0.5633 (10)	0.2803 (6)	0.5351 (8)	0.044 (2)
O2	0.3381 (11)	0.2106 (6)	0.1834 (8)	0.038 (2)
H2O	0.2428	0.2147	0.1437	0.08 (6)*
N1	0.2813 (13)	0.3302 (7)	0.3748 (9)	0.038 (3)
N2	0.6101 (12)	0.1521 (7)	0.3455 (9)	0.033 (3)
C1	0.4697 (16)	0.3364 (10)	0.5848 (13)	0.050 (4)
H1A	0.5391	0.3755	0.6457	0.06*
H1B	0.4074	0.3021	0.6276	0.06*
C2	0.3579 (18)	0.3894 (9)	0.4808 (13)	0.048 (4)
H2A	0.2761	0.4173	0.5113	0.057*

H2B	0.4178	0.4349	0.453	0.057*
C3	0.1879 (19)	0.3814 (10)	0.2666 (13)	0.061 (5)
H3A	0.0986	0.4086	0.2864	0.092*
H3B	0.1488	0.3434	0.1954	0.092*
H3C	0.2551	0.4259	0.247	0.092*
C4	0.1717 (17)	0.2652 (10)	0.4069 (14)	0.052 (4)
H4A	0.0854	0.2955	0.427	0.079*
H4B	0.2304	0.2311	0.4781	0.079*
H4C	0.1284	0.2271	0.3365	0.079*
C5	0.4044 (16)	0.1367 (9)	0.1424 (13)	0.046 (4)
H5A	0.3869	0.1398	0.0523	0.055*
H5B	0.3528	0.0839	0.1607	0.055*
C6	0.5814 (15)	0.1335 (9)	0.2087 (11)	0.038 (3)
H6A	0.6234	0.0759	0.198	0.045*
H6B	0.637	0.1766	0.1725	0.045*
C7	0.7829 (17)	0.1630 (11)	0.4054 (13)	0.059 (5)
H7A	0.8063	0.1534	0.4942	0.089*
H7B	0.8144	0.2215	0.3905	0.089*
H7C	0.8413	0.1214	0.3707	0.089*
C8	0.5534 (19)	0.0790 (9)	0.4089 (13)	0.054 (4)
H8A	0.5948	0.0246	0.3877	0.08*
H8B	0.4381	0.0775	0.382	0.08*
H8C	0.5906	0.0876	0.4982	0.08*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0801 (9)	0.0630 (7)	0.0579 (7)	−0.0279 (7)	0.0159 (6)	0.0090 (6)
Cu1	0.0337 (9)	0.0659 (13)	0.0304 (9)	0.0154 (9)	−0.0015 (7)	−0.0125 (9)
O1	0.040 (5)	0.057 (6)	0.032 (5)	0.016 (5)	0.006 (4)	−0.003 (5)
O2	0.030 (6)	0.046 (6)	0.034 (5)	−0.003 (4)	0.004 (4)	−0.012 (5)
N1	0.042 (7)	0.047 (7)	0.021 (6)	0.013 (5)	0.005 (5)	−0.001 (5)
N2	0.033 (6)	0.045 (7)	0.020 (5)	−0.010 (5)	0.005 (5)	−0.001 (5)
C1	0.038 (9)	0.063 (11)	0.046 (9)	0.004 (8)	0.005 (7)	−0.006 (8)
C2	0.050 (9)	0.046 (9)	0.045 (8)	0.003 (8)	0.010 (7)	−0.015 (8)
C3	0.073 (12)	0.062 (11)	0.037 (8)	0.027 (9)	−0.001 (8)	−0.001 (8)
C4	0.048 (9)	0.070 (11)	0.053 (9)	−0.019 (8)	0.035 (8)	−0.008 (8)
C5	0.053 (9)	0.048 (9)	0.032 (8)	0.011 (7)	0.007 (7)	0.005 (7)
C6	0.038 (8)	0.045 (9)	0.030 (7)	0.011 (7)	0.011 (6)	0.014 (6)
C7	0.059 (10)	0.071 (12)	0.042 (9)	0.033 (9)	0.008 (8)	−0.009 (9)
C8	0.081 (12)	0.043 (9)	0.034 (8)	0.000 (8)	0.013 (8)	0.011 (7)

Geometric parameters (Å, º)

I1—Cu1	2.928 (2)	C2—H2B	0.97
Cu1—O1	2.030 (9)	C3—H3A	0.96
Cu1—N2	2.058 (11)	C3—H3B	0.96
Cu1—N1	2.059 (10)	C3—H3C	0.96

Cu1—O2	2.010 (8)	C4—H4A	0.96
O1—C1	1.399 (16)	C4—H4B	0.96
O2—C5	1.400 (16)	C4—H4C	0.96
O2—H2O	0.82	C5—C6	1.502 (17)
N1—C3	1.466 (15)	C5—H5A	0.97
N1—C2	1.481 (16)	C5—H5B	0.97
N1—C4	1.488 (16)	C6—H6A	0.97
N2—C7	1.465 (16)	C6—H6B	0.97
N2—C8	1.476 (17)	C7—H7A	0.96
N2—C6	1.495 (15)	C7—H7B	0.96
C1—C2	1.512 (19)	C7—H7C	0.96
C1—H1A	0.97	C8—H8A	0.96
C1—H1B	0.97	C8—H8B	0.96
C2—H2A	0.97	C8—H8C	0.96

O1—Cu1—N2	93.9 (4)	N1—C3—H3A	109.5
O1—Cu1—N1	82.2 (4)	N1—C3—H3B	109.5
N2—Cu1—N1	155.4 (4)	H3A—C3—H3B	109.5
O1—Cu1—I1	101.0 (3)	N1—C3—H3C	109.5
N2—Cu1—I1	101.3 (3)	H3A—C3—H3C	109.5
N1—Cu1—I1	103.3 (3)	H3B—C3—H3C	109.5
I1—Cu1—O2	99.6 (3)	N1—C4—H4A	109.5
O1—Cu1—O2	159.5 (4)	N1—C4—H4B	109.5
O2—Cu1—N1	92.9 (4)	H4A—C4—H4B	109.5
O2—Cu1—N2	82.3 (4)	N1—C4—H4C	109.5
C1—O1—Cu1	113.3 (7)	H4A—C4—H4C	109.5
C5—O2—H2O	109.5	H4B—C4—H4C	109.5
C3—N1—C2	110.0 (11)	O2—C5—C6	109.0 (11)
C3—N1—C4	108.1 (11)	O2—C5—H5A	109.9
C2—N1—C4	112.7 (11)	C6—C5—H5A	109.9
C3—N1—Cu1	115.2 (9)	O2—C5—H5B	109.9
C2—N1—Cu1	103.5 (8)	C6—C5—H5B	109.9
C4—N1—Cu1	107.4 (8)	H5A—C5—H5B	108.3
C7—N2—C8	108.0 (11)	N2—C6—C5	109.7 (10)
C7—N2—C6	109.3 (10)	N2—C6—H6A	109.7
C8—N2—C6	111.3 (10)	C5—C6—H6A	109.7
C7—N2—Cu1	115.2 (9)	N2—C6—H6B	109.7
C8—N2—Cu1	109.4 (8)	C5—C6—H6B	109.7
C6—N2—Cu1	103.6 (7)	H6A—C6—H6B	108.2
O1—C1—C2	110.1 (11)	N2—C7—H7A	109.5
O1—C1—H1A	109.6	N2—C7—H7B	109.5
C2—C1—H1A	109.6	H7A—C7—H7B	109.5
O1—C1—H1B	109.6	N2—C7—H7C	109.5
C2—C1—H1B	109.6	H7A—C7—H7C	109.5
H1A—C1—H1B	108.2	H7B—C7—H7C	109.5
N1—C2—C1	108.9 (11)	N2—C8—H8A	109.5
N1—C2—H2A	109.9	N2—C8—H8B	109.5
C1—C2—H2A	109.9	H8A—C8—H8B	109.5

N1—C2—H2B	109.9	N2—C8—H8C	109.5
C1—C2—H2B	109.9	H8A—C8—H8C	109.5
H2A—C2—H2B	108.3	H8B—C8—H8C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O1^{i}	0.82	1.68	2.482 (12)	167

Symmetry code: (i) $x-1/2, -y+1/2, z-1/2$.